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Australian Standard 2678.1—1984

METHODS FOR THE ANALYSIS OF ZINC CONCENTRATES

Part 1—DETERMINATION OF THE ZINC CONTENT OF ZINC CONCENTRATES AND FUMES (TITRIMETRIC SOLVENT EXTRACTION METHOD)

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The following interests are represented on Committee MN/5:

Australasian Institute of Mining and Metallurgy

Australian Mineral Development Laboratories

Australian Mining Industry Council

CSIRO, Institute of Earth Resources

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PREFACE

This standard was prepared by the Association's Committee for Copper, Lead and Zinc Ores and Concentrates under the direction of the Minerals Standards Committee. It has been prepared at the request of the producers and users of zinc concentrates in order to provide a method which can be specified in contracts.

The committee organized an interlaboratory test program to obtain information on the repeatability and reproducibility of the method. The following laboratories participated in the test program to provide the data given in Table 1:

Broken Hill Associated Smelters Pty Ltd
Dept of Mineral Resources, N.S.W.
Electrolytic Zinc Co. of A/asia Ltd
Mount Isa Mines
North Broken Hill Limited
Sulphide Corporation Pty Ltd
Woodlawn Mines
Zinc Corporation Ltd

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STANDARDS ASSOCIATION OF AUSTRALIA

Australian Standard
METHODS FOR THE ANALYSIS OF ZINC CONCENTRATES

PART 1—DETERMINATION OF THE ZINC CONTENT OF ZINC CONCENTRATES AND FUMES (TITRIMETRIC SOLVENT EXTRACTION METHOD)

1 SCOPE. This standard sets out an EDTA titrimetric method for the determination of the zinc content of zinc concentrates and zinc oxide fumes.

2 APPLICATION. The method is applicable to zinc concentrates and zinc oxide fumes containing 30 percent to 70 percent Zn. It has been shown that there is no interference by up to 6 percent Cd, 8 percent Mn, 30 percent Cu or 40 percent Pb.

3 REFERENCED DOCUMENTS. The following standards are referred to in this standard:

AS 2164	One-mark Volumetric Flasks
AS 2165	Burettes and Bulb Burettes
AS 2243	Safety in Laboratories Part 2—Chemical
AS 2407	Separating Funnels and Dropping Funnels
AS XXXX*	Copper, Lead and Zinc Sulphide Concentrates—Determination of Hygroscopic Moisture in the Analysis Sample—Gravimetric Method
BS 4237	Report on Reproducibility of Methods of Chemical Analysis Used in the Iron and Steel Industry

4 PRINCIPLE. The sample is dissolved in mixed acid and bromine. The zinc thiocyanate complex is extracted into methyl iso-butyl ketone. The zinc is re-extracted into an aqueous phase and the mixed solvent aqueous phase titrated with EDTA using a mixed indicator of eriochrome black T and dimethyl yellow.

5 REAGENTS.

5.1 General requirements. During the analysis, only reagents of recognized analytical grade and only distilled water or water of equivalent purity shall be used.

NOTE: A number of the reagents used in this method (bromine, potassium cyanide, perchloric acid) are toxic or dangerous. Advice on procedures for handling and disposal of these reagents is contained in AS 2243, Part 2.

5.2 SOLIDS.

5.2.1 Sodium carbonate anhydrous.

5.2.2 Tri-sodium citrate dihydrate.

5.2.3 Ascorbic acid.

5.2.4 High purity zinc metal (99.99 percent zinc). The surface of the metal must be free from oxide prior to use, and may be cleaned by immersing the metal in hydrochloric acid (ρ_{20} 1160 kg/m^3, diluted 1 + 9) for 1 min, washing well with water followed by acetone and drying in an oven at 50°C.

5.3 Solutions.

5.3.1 Hydrochloric acid (ρ_{20} 1160 kg/m^3).

5.3.2 Hydrochloric acid (500 mL/L). Add 500 mL of hydrochloric acid (5.3.1) to 500 mL of water.

5.3.3 Hydrochloric acid (50 mL/L). Dilute 50 mL of hydrochloric acid (5.3.1) to 1 L with water.

5.3.4 Nitric acid (ρ_{20} 1410 kg/m^3).

5.3.5 Hydrofluoric acid (ρ_{20} 1130 kg/m^3).

5.3.6 Perchloric acid (ρ_{20} 1700 kg/m^3).

5.3.7 Ammonia solution (500 mL/L). Add 500 mL of ammonia (ρ_{20} 880 kg/m^3) to 500 mL of water.

5.3.8 Bromine.

5.3.9 Iron(III) chloride solution (600 g/L). Dissolve 60 g of iron(III) chloride.6-water ($FeCl_3.6H_2O$) in water and dilute to 100 mL.

5.3.10 Thiourea solution (80 g/L). Dissolve 80 g of thiourea in 1 L of water.

*In course of preparation.